Generation of Large-area, Crack-free GaN layers on Si Substrates

Final Report

THOMAS F. KUECH
UNIVERSITY OF WISCONSIN

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UNIVERSITY OF WISCONSIN
DEPARTMENT OF CHEMICAL ENGINEERING
14151 ENGINEERING DRIVE
MADISON, WI 53706

Summary:

The development of truly compliant layers, compatible with Si-based tooling, was investigated for the growth of GaN on a Si substrate. For this project, thin Si layers were fabricated on a metal bonding layers. The metal bonding medium was chosen such that it would be liquid at the GaN growth temperature and hence be compliant, allowing lateral motion of the Si template layer as growth proceeded and during the cooling of the grown structure from the growth temperature. The stability of thin Si substrate layer during thermal cycling was determined both experimentally and through modeling. Constraints on the formation of metal bonded structures based on the interplay of the elastic strain energy, ε_{ES}, and bond energy, Γ_{BE}, of a template/DB multi-layer structure supported on a metal layer were developed.

Technical Report:

Metal bonding layers are being investigated as bonding media for several materials integration applications.[1-4] For example, metal bonding is being investigated as a means of achieving high bond strength at temperatures lower than conventional Si hydrophilic bonding.[1-3] Conventional Si wafer bonding requires annealing temperature of 1000°C to develop a strong covalent bond.[5] For applications involving electrical devices this anneal temperature is too high to maintain the designed dopant profiles or the integrity of deposited metal interconnect layers. Thermo-compression bonding with Au and Al metal layers has been investigated as a means to obtain strong bonding without the requirement of high temperature anneals.[1-2] An electrically conductive bonding layer also can provide a path to 3D integration of electronic devices.[4]

Metal bonding layers have additionally been used in the development of compliant substrates for lattice mismatched heteroepitaxy.[4] For example, a semiconductor layer, patterned on the back side, bonded to another wafer for mechanical support via a metal layer has been previously studied.[4] This patterned wafer is thinned to 100-200 nm before epitaxial growth on the thin layer is performed. During epitaxial growth, the metal melts and becomes a liquid layer so the thin template layer is able to approximate a freestanding thin film. The strain associated with lattice-mismatched heteroepitaxy can be partitioned between the thin template and growing film, independent of the thick mechanical support.[4] The substrates were successfully fabricated and used in epitaxial growth, however, the observed modifications to the epitaxial layer growth were never fully understood.[4,5]

Another potential application of metal bonded structures is in the mitigation of crack formation during heating and cooling of thermally mismatched semiconductor layers. For example, when GaAs is bonded to Si the temperature range for subsequent processing steps is limited due to the thermal expansion coefficient (TEC) mismatch between the layers.[6,7] The limit on the cycling temperature can be alleviated somewhat by thinning one layer since the thinner layer can more readily conform to the thicker layer.[6,7] Bonding of the TEC mismatched layers with a liquid layer would mechanically separate the layers and allow each layer to expand and contract freely. Fabricating a structure with a liquid layer is difficult to implement but can be approximated by the use of a low melting point metal that is liquid over a significant temperature range. In this case, the stress that develops in a TEC mismatched bonded structure is only due to the temperature range where the metal is solid. If a low melting temperature metal such as In is used the temperature regime where TEC strain is generated is only between room temperature and 156°C. Low melting point metal-bonded structures would also be interesting for

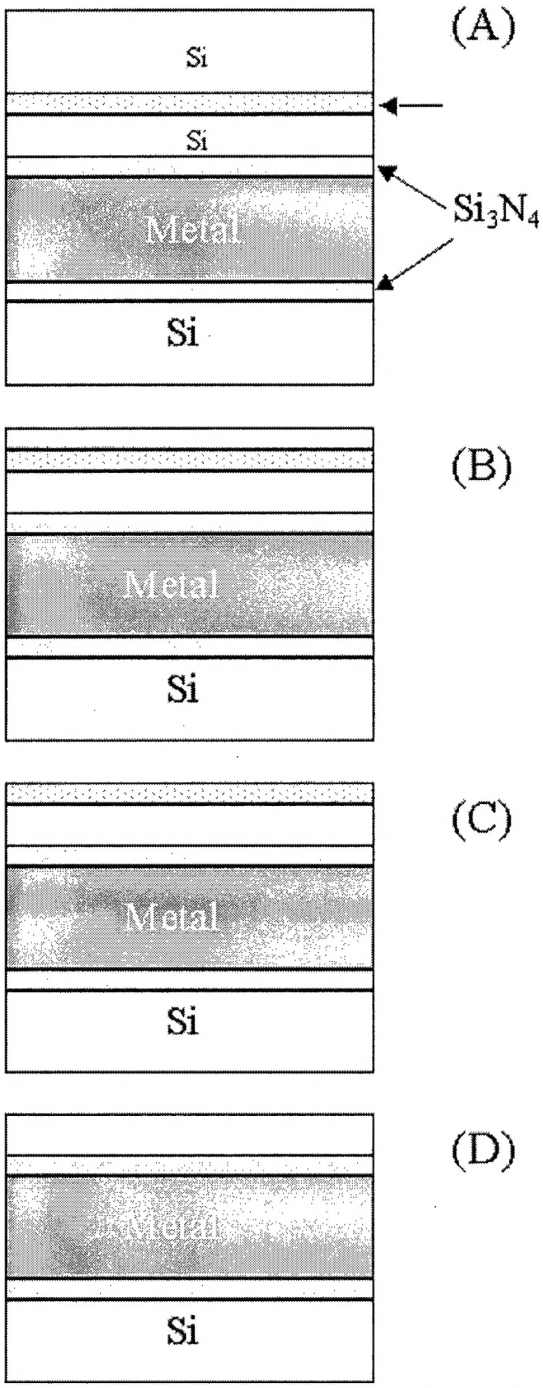

Figure 1: Schematic of the metal bonded structure formation. (A) shows the as bonded structure. (B) is after mechanical grinding down to within 40 mm of the bonded interface. (C) is after etching in TMAH solution. (D) is after the final 10% HF dip to remove the oxide from the BOX region of the SOI wafer.

integrating GaN on Si, another highly thermally mismatched system. A thin Si layer, a template layer for crystal growth, supported on a mechanical handle wafer via a low melting point metal, such as In, would provide a means of floating the GaN on the mechanical host wafer, mechanically decoupling the layers and reducing the stress in the GaN caused by thermal cycling.

The ability to form thin layers under strain on liquid metals has not been addressed. The focus of this study was on a multi-layer structure consisting of a semiconductor layer (template) coated with a diffusion barrier (DB). This general structure would prevent the diffusion and reaction of metal with the template layer, perhaps because of electrical devices in the template layer.

In this investigation In, Al, Al/Si, and In/Au/Ti metal bonding layers were used to form Si-based structures consisting of 2 μm template layers. Issues involving the bond strength and structure stability are presented and addressed in terms of a balance between the strain energy stored in the template layer/DB multi-layer and the interfacial energy between the liquid metal and the strained-multilayer. The morphology of the 2 mm Si template layer for these metal-bonded structures was investigated.

Structure Formation

The sequence for forming metal bonded structures is detailed in Figure 1. The substrates used in the experiments were 4″ (100) Si wafers as well as 4″ SOI substrates. The commercial SOI wafers consisted of a 2 ± 0.5 μm (111) top Si wafer on a 0.5 ± 0.04 μm buried oxide layer on (100) bulk Si layer. Prior to diffusion barrier or metal deposition, wafers were dipped in a H_2O: HF (50:1) solution for 20 s to remove the native oxide on the silicon surface, followed by a deionized water rinse. The DB used in each of these experiments was a 100 nm Si_3N_4 layer deposited by plasma enhanced chemical vapor deposition (PECVD) at 350°C. Both the SOI

and bulk Si wafers were coated with the DB layer. Some structures were formed with a thermal SiO_2 layer 50 nm thick.

Both wafers were coated with metal prior to bonding. Several metal layers were employed during these investigations. In, Al, Al/Ge, and Ti/Au/In were investigated. In, with a low melting point, T_{melt}=155°C, and γ_{metal} of 550 erg/cm^2 was chosen because of the low melting point. Ti/Au/In was investigated as a means of improving the wetting behavior of In on the DB. The addition of Ti/Au layers is an established method for improving the wetting of In on surfaces. Al with γ_{metal} of 480 erg/cm^2 was also used. The contact angle of Al on Si_3N_4 has been reported as a function of temperature and more readily wets Si_3N_4 than In.[13] Al/Ge was investigated because the Al/Ge eutectic has a lower melting point than Al, providing a greater region where this metal composition is molten.

For the In metal-bonded substrates, a 4 μm In metal layer was deposited by thermal evaporation on both the SOI wafers and the Si handle wafer. The samples were rinsed with megasonically agitated DI water prior to loading into the wafer bonder. The chamber was evacuated to ~10^{-4} mbar. Wafer bonding was performed by touching the wafers together in the center and allowing the bonding front to propagate out to the edges. The bond was strengthened by applying a 700 N force to the wafer pair while heating to 80°C. After the initial bond, the samples were then annealed in N_2 for 30 minutes at 260°C.

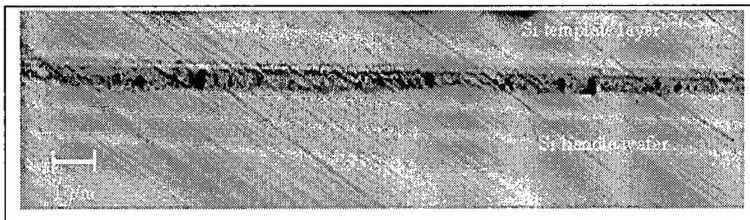

Figure 2: An SEM image of a metal-bonded substrate using an Al metal layer. In this case the Si template layer is 2 μm thick. The relative stability of the Si template is apparent. An Al interaction with the Si handle wafer is also obvious in the bottom image.

The above description generally describes the process that was used for each metal in these structures. The largest difference between each metal layer was the anneal temperature in N_2 for 30 minutes after the initial room temperature bond. In each case, a temperature ~200°C above the melting point was selected. In each substrate structure, therefore, the bond was strengthened from the initial room temperature bond by molten metal bonding of the two wafers. The bonded wafer pair was mechanically thinned down from the SOI substrate to ~40 μm approaching the bonding interface as shown in Figure 1. The remaining (100) Si was etched using a 25 wt.% tetramethylammonium hydroxide (TMAH) solution kept in a thermal bath of 80°C, which has a selectivity of (100) Si-to-SiO_2 greater than 9000.[12] After etching, the substrates were diced into 1 x 1 cm^2 pieces. The integrity of the fabricated structures were analyzed using optical microscopy and in detailed cross section using scanning electron microscopy (SEM).

Results from the characterization of Bonded Structures

Metal-DB Interfacial Energy Impact on Structure Formation

The metal bonded structures were all fabricated by heating the structures above the metal melting point in an attempt to obtain a strong bond. Therefore, the relationship between the DB and the molten metal is an important issue. The bond energy of the molten metal-DB interface, Γ_{BE}, can be determined from the energy difference between two free surfaces, one for the DB interface and one for the metal layer, and the new metal-DB interface.[10] Specifically,

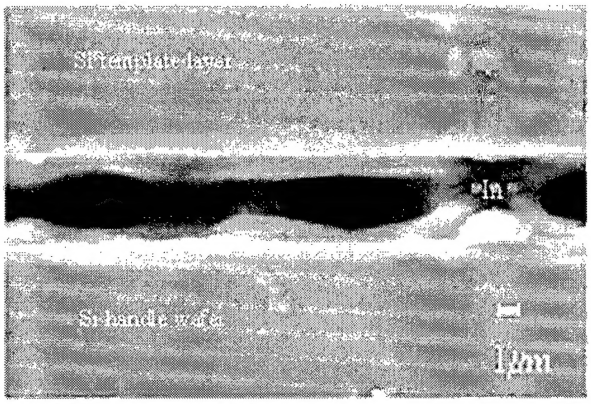

Figure 3: An SEM image of a metal-bonded substrate using an In metal layer. In this case the Si template layer is ~10 μm thick. The balls of In on the surface are readily apparent.

$$\Gamma_{BE} = \gamma_{DB} + \gamma_{metal} - \gamma_{DB/metal} \qquad [1]$$

where γ_i is the interfacial energy of the layer. When the metal layer is a liquid, it is possible to apply Young's equation that defines a contact angle, θ, between three phases,

$$\gamma_{DB} = \gamma_{DB/metal} + \gamma_{metal}\cos(\theta) \qquad [2]$$

Simply combining equations (1) and (2) permits the usual expression for the bond energy between the DB and the metal in terms of the surface energy of the metal and the contact angle with the DB,

$$\Gamma_{BE} = \gamma_{metal}(1+\cos(\theta)) \qquad [3]$$

Equation (3) can be used to understand trends for Γ_{BE} based on the materials properties used in the fabrication of metal-bonded structures. Maximizing Γ_{BE}, hence maximizing the structure stability, requires a metal with a high interfacial energy, γ_{metal}, while also possessing a low contact angle, θ.

This equation provides a rapid evaluation of the metal to be used in the metal bonded structure since both the interfacial energy and contact angle on standard semiconductor materials are available. For example, Al has an interfacial energy, γ_{metal}, of 480 erg/cm^2.[11] The contact angle of Al on Si_3N_4 is reported to be 90° at 1050°C. This suggests, therefore, that Γ_{BE} with this combination of materials would be 438 erg/cm^2 in this temperatutre region. This trend is supported by SEM observation of Al-metal bonded structures. Figure 2 presents a cross section image of Si-based metal bonded structure using Al. The interface is clearly flat and appears to be well-bonded because molten Al and the Si_3N_4 DB layer have a favorable interaction.

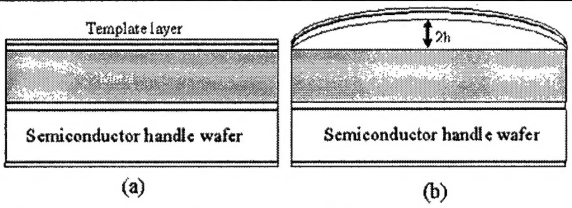

Figure 4: (A) shows a well-bonded metal-bonded substrate. (B) is a schematic of one simplified form of debonding that occur where the DB (□)/Si template (□) pulls away from the metal (■) layer due to elastic strain energy stored in the DB/Si template multi-layer.

The impact of the molten metal-DB interface is evident in Figure 3. Figure 3 is an SEM cross section image of an In-metal bonded structure. As shown in this image the Si layer is relatively flat despite the agglomeration or balling of the In at the bonded interface. The In does not wet the DB in this case. The contact angle for the In balls can be approximated using this SEM at ~160°. According to equation (3), Γ_{BE} is 33 erg/cm^2 at the DB molten In bonded interface. This bond energy is low and suggests an area of concern for In-based metal bonded structures despite the metals favorable properties such as low melting point. In can be used in forming metal-bonded structures by employing a wetting layer of Au/Ti. In this investigations, the addition of Au/Ti improved the bonding using In metal, however, metallic phase transitions impacted the structure reliability as will be discussed in later a later section.

DB-Template Layer Curvature Impact on Structure Stability

For the In metal-bonded structures, the Si template thickness was straightforwardly thinned to 10 μm. As the template was thinned towards the 2 μm proposed for the metal-bonded structure, however, the samples debonded as noted by visual inspection of the structure. The debonding of In-only metal bonded structures can be attributed to the agglomeration or balling of In during the molten metal bonding process. However, despite this balling structures consisting of 10 μm Si template layers were fabricated. This result can be understood in terms of the role of the DB/template layer strain in metal-bonded structure stability.

The model structure is shown schematically in Figure 4(a). A semiconductor layer, the template, coated with a diffusion barrier (DB) on one side only is supported on a mechanical handle wafer via a liquid metal layer. The handle wafer is coated symmetrically to rule out thermal strains and to ensure the handle wafer is flat at all times. Common types of DB layers are SiO_2 and Si_3N_4. For many types of applications, including heteroepitaxial growth on this structure, the template/DB multi-layer structure must remain flat. Intrinsic stresses in the template/DB multi-layer result in a tendency for this multi-layer to bend or buckle during structure formation. The energy needed to maintain a flat layer from an equilibrium-curved shape is derived from a calculation of the elastic strain energy, ε_{ES}. Requirements to maintain a bonded interface have been considered for a radial model in terms of the elastic strain energy that must be overcome to close a gap of height 2h over a lateral dimension of 2R.[8] For the case when the lateral dimension is much larger than the gap spacing, the value of ε_{ES} that must be overcome is given by the expression,

$$\varepsilon_{SE} = \frac{2.4h^2 M_1 d_1^3 M_2 d_2^3}{R^4 (M_1 d_1^3 + M_2 d_2^3)} \qquad [4]$$

where M_i are the moduli of the layers and d_i are the thickness.[8] The case that is being considered in this investigation is for curvature induced in the template/DB multi-layer structure that could cause a gap to develop, as shown in Figure 4(b). Since the handle wafer consists of a thick semiconductor material and the metal layer is at most several microns thick, the metal/bulk wafer were assumed to have the properties of the handle wafer. Ignoring other complications to bonding phenomena, such as surface roughness, the gap that must be closed to maintain a bonded interface is strictly a result of the DB/template curvature. Due to the orders of magnitude difference in thickness,

$$M_{Si} d_{Si}^3 + M_{DB/template} d_{DB/template}^3 \approx M_{Si} d_{Si}^3 \qquad [5]$$

Therefore, equation (4) can be simplified to,

$$\varepsilon_{SE} = \frac{2.4h^2 M_{DB/template} d_{DB/template}^3}{R_{wafer}^4} \qquad [6]$$

where R has been replaced by R_{wafer} since the curvature occurs over the whole wafer. The template layer thickness considered in our experiments was on the order of 1-5 μm and the DB thickness is 0.03-0.5 μm, the M_i and d_i for equation (6) were approximated as that of the template layer. The distinction between the template and DB layers becomes critical in the calculation of the curvature of the template/DB multi-layer structure.

When $d_{template}$ and d_{DB} become comparable, the 2nd order Stoney's equation must be employed. The 2nd order Stoney's equation expresses the radius of curvature, $R_{curvature}$, of the DB/ template mutli-layer in terms of the stress in the DB, the thickness of the DB and template and the moduli of the layers,[9]

$$\frac{1}{R_{curvature}} = 6\sigma_{DB}\frac{d_{DB}}{d_{template}}\frac{1}{M_{template}} + 6\sigma_{DB}\frac{d_{DB}^2}{d_{template}^3}\frac{(M_{template} - 4M_{DB})}{M_{template}^2} \quad [7]$$

where σ_{DB} is the stress in the DB, h_i is the thickness of layer i, and M_i is the modulus of layer i.[9] The 2nd order correction to the Stoney equation was used for the case where the thickness of the template and DB became comparable.

The gap height, h, that must be closed becomes a matter of geometry involving the radius of curvature of the structure and the radius of the wafer. Assuming that $R_{curvature}$ is always much larger than R_{wafer} permits approximating the gap spacing by,

$$2h = \frac{R_{wafer}^2}{4R_{curvature}} \quad [8]$$

The combination of equations 6, 7, and 8 permitted the calculation of the elastic strain energy that must be overcome to maintain a flat structure from the DB/template multi-layer structure. Values for the base case are calculated using the model parameters presented in Table 1. The DB is considered to be Si_3N_4 and the template layer is (111) Si. Although this model can handle any type of material, these materials were chosen as the base case due to their individual importance. The stress in the Si_3N_4 layer provided in Table1 was measured for plasma enhanced chemical vapor deposited (PECVD) SiN_y layers based on the measured Si wafer radius of curvature change after SiN_y deposition.

Figure 5 is a plot demonstrating the changes in ε_{ES} as a function of the template layer thickness for the base case using materials parameters provided in Table 1. There is a maximum in the elastic strain energy required to maintain a flat structure as the template layer thickness decreases for a given DB layer thickness. This maximum occurs because of the increased curvature that results from the stress partitioning between the DB and the template layer. As the template layer becomes thinner the maximum stress initially in the DB now relocates and resides within the template layer. The location of this maximum is important to note. For this particular case, assuming 200 nm of Si_3N_4 and (111) Si template, the maximum in the elastic strain energy occurs at ~2 μm of Si. This result, therefore, describes the results observed from In-based metal bonded structures. Despite the obviously poor bonding achieved as a result of In agglomeration the structure remains well bonded to Si template layer thickness ~10 mm because the strain energy is still low. However, when the Si template layer is thinned further ε_{ES} increases sharply as shown in Figure , leading to structure debonding. These model equations permit evaluation of the general trends expected for the metal-bonded structure stability as a function of the dimensions and materials properties desired.

Figure 5 also presents the effect of the DB layer thickness on ε_{ES}. As the DB layer thickness decreases, the peak in the elastic strain energy shifts to thinner template layers and decreases in absolute magnitude. The thinnest possible DB layer, within the constraints of the application, is needed to minimize the elastic strain energy.

Table 1: Material and Structural Parameters Used to Determine ε_{ES} for the Base Case.

Parameter	Typical Value Range
h_{DB}	200 nm
M_{DB} (Si_3N_4)	$2.5x10^{11}$ Pa
h_{Si}	1-500 μm
$M_{(111)Si}$	$2.290x10^{11}$ Pa
σ_{DB}	$1x10^{9}$ Pa
D_{wafer}	10.1 cm

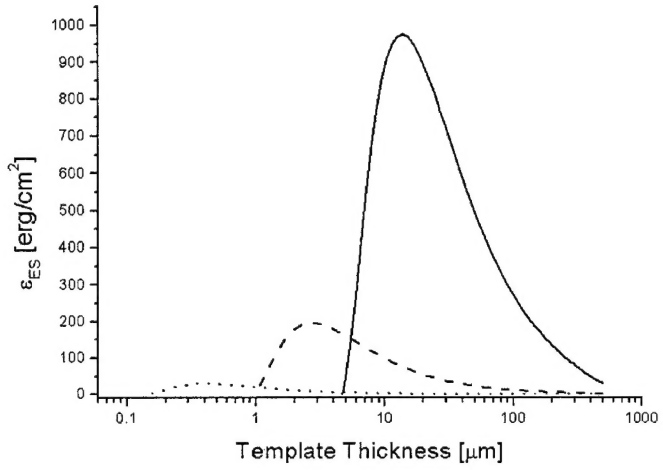

Figure 5: ε_{ES} as a function of template layer thickness; (—) is for 1000 nm of DB, (---) is for 200 nm of DB, (···) is for 20 nm of DB. In all cases, the σ_{DB} is 1 GPa and M_{DB} is $2.5x10^{11}$ Pa.

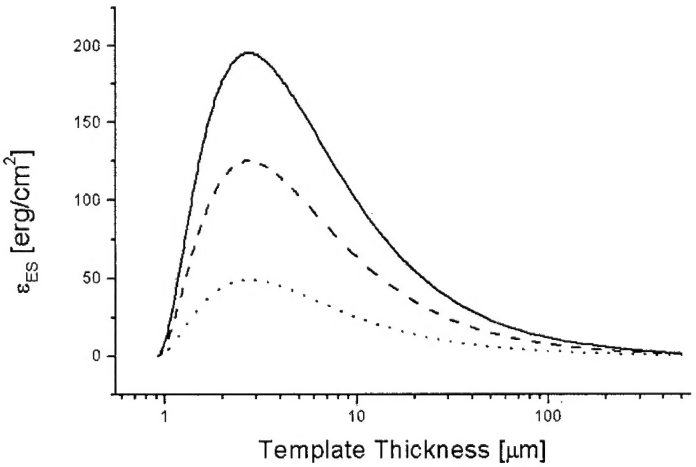

Figure 6: ε_{ES} as a function of template layer thickness; (—) is for 1000 Mpa stress, (---) is for 800 Mpa stress, (···) is for 500 MPa. In all cases, the thickness of the DB is 200 nm and M_{DB} is $2.5x10^{11}$ Pa.

The magnitude of the DB stress, σ_{DB}, was also evaluated. Figure 6 shows the change in ε_{ES} as a function of the template layer thickness for different stress levels in the DB. All other materials parameters were the same as the base case given in Table 1. The σ_{DB} changes the absolute magnitude of the elastic strain energy while the DB thickness corresponding to the maximum remains the same. More specifically, as σ_{DB} increases, the elastic strain energy required to keep the template/DB multi-layer structure flat also increases. This result suggests that altering the DB layer to a material with the lowest possible stress will improve the structureal stability with respect to the tendency to bend or buckle while resting on the liquid bonding layer. Although Si_3N_4 is an excellent diffusion barrier, it has a larger stress than other possible DB layers such as SiO_2. To maximize the DB effectiveness, a balance between the diffusion rate of the metal through the DB and the stress caused by the DB must be found.

Other factors that influence the elastic strain energy are that a stiffer template layer, larger M_i, lowers ε_{ES} as does lower value of M_{DB}. These parameters can be controlled by choice of the fabrication materials and

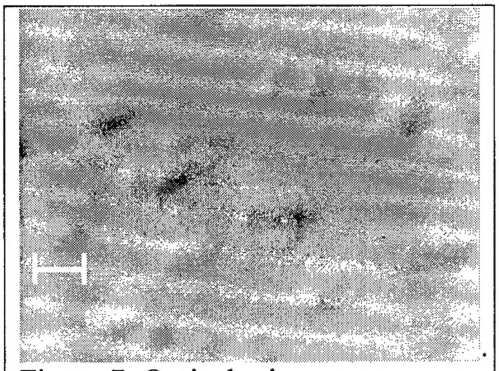

Figure 7: Optical microscopy images at 200X magnification (50μm marker) of the Si template made with a Al metal bonding layer. This structure was fabricated with molten metal bonding at 700°C after the initial room temperature bond.

processes used in the formation of the metal-bonded structure.

The Al, unlike the In metal bonded structures, readily wets the Si_3N_4 DB as has been reported.[13] This metallurgy lead to a Si metal-bonded structure that remained bonded through all stages of fabrication. Although the structure remained bonded, there is a fine scale roughness observed on the surface of the Si template layer by optical microscopy as shown in Figure 7. This fine scale roughness may be a result of the fabrication process, such as trapped interfacial gas, or a result of an inherent instability in thin layers supported on liquid layers that has not been accounted for in the preliminary development of expressions for Γ_{BE} and ε_{ES}. In addition, local variations in the DB strain may also impact the template layer morphology. Si_3N_4 has been demonstrated to have local variations in the strain according to previous reports. In order to obtain flat, metal bonded structures suitable for epitaxial growth, investigations of the origin of this fine-scale roughness should be continued.

The final structure, shown in Figure 8, illustrate many of the instabilities discussed above. We have investigated the use of In, Ti/Au/In and Al as bonding media for the formation of Si substrates for subsequent GaN growth at elevated temperatures. A ~2μm Si layer with an intermediary SiO_2 or Si_3N_4 diffusion barrier, is bonded to a handle wafer via a low melting point metal or metal alloy layer. The substrate is intended to mitigate the thermal expansion mismatch induced stresses that otherwise results in the cracking of the GaN films grown on conventional Si substrates upon cooling from the ~1100°C GaN growth temperature. The interfacial energy between the metal and the diffusion barrier, the robustness of the diffusion barrier, and metallurgical reactions within the bonding media determine the extent to which the upper Si layer remains planar at elevated temperatures. In-bonded structures with SiO_2 diffusion barriers

Figure 8: Cross section SEM of Ti/Au/In metal structure. A 2 μm Si template layer is bonded to a Si handle wafer using an SiO_2 DB and the Ti/Au/In metal bonding layer. Cracking and instability in the 2 μm layer is visible.

were not stable at the elevated temperatures due to the high interfacial energy between the In and SiO_2. The morphology of Ti/Au/In bonded structures after temperature cycling is due to silicide formation and the reaction between Au and In. The Al-bonded structures without a bottom SiO_2 diffusion barrier between the metal and handle wafer proved more robust during temperature cycling to 1100°C.

Summary of constraints on the metal-bonded compliant structure.

Constraints on the formation of metal bonded structures based on the interplay of the elastic strain energy, ε_{ES}, and bond energy, Γ_{BE}, of a template/DB multi-layer structure supported on a metal layer were considered. Based on these equations it was found that there is a peak in the elastic strain energy as the template layer is thinned. The elastic strain energy was also found to increase with DB layer stress and DB thickness, indicating general guidelines for structure engineering. The equation for the bond energy indicated that metal layers with a high interfacial energy and low contact angles are best choice. These results were supported by fabrication of metal bonded structures with a Si template and In and Al based metallurgy. The In metal-bonded structures demonstrated agglomeration or balling of In at the metal/DB interface as a result of the molten-metal bonding process. This agglomeration was not noted for more favorable contact angles from different metal bonding layers for example Al or In/Au/Ti. 2 μm Si template layers were fabricated on metal bonded layers, however, further investigation of the stability of metal bonded structures is required as a fine scale roughness of the thin Si template was noted for these types of metal bonded structures.

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Thomas F. Kuech

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University of Wisconsin, Department of Chemical Engineering
1415 Engineering Drive, Madison, WI, 53706

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13. ABSTRACT (Maximum 200 words)

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